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IS 2305 (1988): Method for mercurous nitrate test for copper and copper alloys [MTD 8: Copper and Copper Alloys]



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Indian Standard
METHOD FOR
MERCUROUS NITRATE TEST FOR COPPER
AND COPPER ALLOYS
(First Revision)

1. Scope — This standard prescribes the method for conducting mercurous nitrate test for wrought copper and copper base alloy products. It is an accelerated test for the purposes of detecting the presence of residual (internal) stress that might bring about failure of the material in service or storage through stress corrosion cracking.

1.1 While this method has also been used for testing assemblies and partial assemblies, it is not intended for that purpose and some modification may be required for such use.

2. Terminology — For the purpose of this standard, the following definitions shall apply.

2.1 Stress Corrosion Cracking — Spontaneous failure of metals by cracking under combined action of corrosion and stress, residual or applied.

2.2 Residual Stress — Stress that remains within a body as a result of plastic deformation.

2.3 Applied Stress — Stress that is set up and exists in a body during application of an external load.

3. Mercurous Nitrate Solution — One percent.

3.1 Concentration — The test solution shall be an aqueous mercurous nitrate solution containing 10 g of mercurous nitrate and 10 ml of concentrated nitric acid (sp gr 1.42) per litre of solution.

3.2 Preparation — The solution shall be prepared by either of the following procedures.

3.2.1 Procedure A — Dissolve 11.4 g of mercurous nitrate dihydrate ($\text{HgNO}_3 \cdot 2\text{H}_2\text{O}$) or 10.7 g of mercurous nitrate monohydrate ($\text{HgNO}_3 \cdot \text{H}_2\text{O}$) in approximately 40 ml of distilled water acidified with 10 ml of concentrated nitric acid (sp gr 1.42). After the crystals are completely dissolved, dilute the solution with distilled water to 1 000 ml.

Note — The mercurous nitrate crystals are obtainable in both the monohydrate and dihydrate form and should be handled with caution because of their highly toxic effects. When weighing crystals, the weight of the water of crystallization should be taken into consideration. The mercurous nitrate crystals are photosensitive and when they have turned yellow are difficult to dissolve.

3.2.2 Procedure B — Dissolve 76 g of mercury in 114 ml of dilute nitric acid (1 : 1). Carefully dilute with distilled water to 1 000 ml. This provides a concentration of 100 g of nitric acid after a slight loss due to heating. Add the water in small portions while stirring to prevent local overdilution. This gradual dilution, together with the excess acid, will prevent precipitation of basic salts of mercury. Dilute 100 ml of this solution (10 percent) with 7 ml of concentrated nitric acid (sp gr 1.42) and 893 ml of water.

Note — If heating is used in either of the above procedures, the container should be covered with watch glass to prevent loss of nitric acid and water to the atmosphere.

4. Test Piece

4.1 The length of the test piece shall be at least 150 mm for products of diameter less than or equal to 75 mm. For products of diameter greater than 75 mm, the length shall be subject to agreement between the purchaser and the supplier.

4.2 Care should be taken to avoid grinding stresses during cutting of the test piece. Burrs, if any, should be removed by filing.

4.3 Test pieces for the mercurous nitrate test shall not be marked for identification by stamping.

5. Procedure

5.1 First degrease the test piece. Then totally immerse it in an aqueous solution of sulphuric acid (15 percent by volume) or in a mixture containing 60 parts by volume of distilled water and 40 parts by volume of concentrated nitric acid for a period not exceeding 30 seconds, to remove all traces of carbonaceous matter and oxide films. Remove the test piece from the pickling solution and wash it immediately in running water. Then drain the test piece free of excess water and totally immerse it in the mercurous nitrate solution. Use at least 1.5 ml of test solution per square centimetre of exposed surface of the test piece.

5.2 After 30 minutes, remove the test piece from the mercurous nitrate solution and wash it in running water. Wipe off any excess mercury from the surface of the test piece. Examine the test piece at once, unless otherwise specified in the material specification. The material specification may permit a lapse of time before examination, which may vary with the alloy being tested. In case of doubt regarding the presence of cracks, volatilize (with caution) the mercury on the surface of the test piece by the application of heat on a hot-plate or in an oven. Then examine the specimen for cracks under suitable magnifying equipment at a magnification of 10 to 18 X.

Caution — Mercury is a definite health hazard and, therefore, equipment for the detection and removal of mercury vapour produced in volatilization is recommended. The use of rubber gloves is advisable.

5.3 A solution, the concentration of which is not in accordance with that specified in 3, shall not be used. However, the spent solution can be used after proper replenishment procedure given in Appendix A.

Caution — Mercury is a definite health hazard in use and disposal. The method of mercurous nitrate solution disposal is given in Appendix B.

6. Test Requirements

6.1 The interpretation of the visual appearance of the test pieces after testing is to be specified in the relevant material specification.

APPENDIX A

(Clause 5.3)

REPLENISHMENT PROCEDURE OF SPENT MERCUROUS NITRATE SOLUTION

A-1. The spent solution may be reclaimed by replenishing the mercurous nitrate solution to a 1 percent concentration as follows.

A-1.1 Measure, as accurately as possible in a small graduated cylinder, 50 ml of the spent mercurous nitrate solution.

A-1.2 Transfer the solution to a Erlenmeyer flask and add 10 ml of nitric acid (1 : 1).

A-1.3 Add slowly potassium permanganate solution (1 percent) from a burette with a constant shaking until there is an excess as indicated by the pink colour, which persists for several minutes.

A-1.4 Add ferrous sulphate crystals until the solution, when shaken, becomes clear. Then titrate the solution with 0.1 N potassium cyanide solution (KCN) to the appearance of a reddish brown colour. Repeat the procedure with 50 ml of a standard mercurous nitrate solution (1 percent).

A-1.5 The ratio, R , of the number of ml of potassium cyanide solution required to titrate the spent solution, to the number of ml required to titrate the standard solution, determine the number of ml, X of 10 percent mercurous nitrate in 3 percent nitric acid required to replenish 1 litre of spent solution. Values of R and X for a litre volume are given in Table 1.

TABLE 1 REPLENISHMENT OF SPENT MERCUROUS NITRATE SOLUTION TO 1 PERCENT CONCENTRATION*(Clause A-1.5)*

$$X = 111.1 (1 - R)$$

where

R = fraction of mercury remaining in solution (determined by titration), and*X* = number of ml of mercurous nitrate solution (10 percent) to be added to 1 litre of spent mercurous nitrate solution to raise the concentration of mercurous nitrate to 1 percent.

<i>R</i>	<i>X</i>	<i>R</i>	<i>X</i>
0.10	100.0	0.56	48.9
0.12	97.8	0.58	46.7
0.14	95.5	0.60	44.4
0.16	93.3	0.62	42.2
0.18	91.1	0.64	40.0
0.20	88.9	0.66	37.8
0.22	86.7	0.68	35.6
0.24	84.4	0.70	33.3
0.26	82.2	0.72	31.1
0.28	80.0	0.74	28.9
0.30	77.8	0.76	26.7
0.32	75.5	0.78	24.4
0.34	73.3	0.80	22.2
0.36	71.1	0.82	20.0
0.38	68.9	0.84	17.8
0.40	66.7	0.86	15.6
0.42	64.4	0.88	13.3
0.44	62.2	0.90	11.1
0.46	60.0	0.92	8.9
0.48	57.8	0.94	6.7
0.50	55.6	0.96	4.4
0.52	53.3	0.98	2.2
0.54	51.1		

APPENDIX B*(Clause 5.3)***METHOD FOR DISPOSAL OF SPENT MERCUROUS NITRATE SOLUTION****B-1.** To mercurous nitrate solution, add sodium hydroxide to pH 10-11.**B-2.** Filter precipitated mercury and other heavy metals.**B-3.** Though the filtrate is low in free mercurous or mercuric ions, it must be further treated before disposal.**B-4.** To each litre of filtrate, add two drops (0.1 cm³) of 24 percent ammonium sulphide.**B-5.** After the second filtering, the filtrate may be discarded.**B-6.** The precipitate should be collected and stored with the mercury contaminated test samples and sold to a metal reclaimer.

EXPLANATORY NOTE

This standard was first published in 1962. In this revision, the following modifications have been made:

- a) The procedure have been updated and brought in line with ISO and ASTM standards.**
- b) Appendices A and B giving the replenishment procedure of spent mercurous nitrate solution and method of its disposal have been added.**

In the preparation of this standard, assistance has been derived from the following standards:

- a) ISO 196-1978 Wrought copper and copper alloys — Detection of residual stress — Mercury (I) nitrate test, issued by the International Organization for Standardization (ISO).**
- b) ASTM B 154-82 Method for mercurous nitrate test for copper and copper alloys, issued by the American Society for Testing and Materials, USA.**